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IS 326-15 (1984): Methods of sampling and test for natural and synthetic perfumery materials (Part 15) Detection of petroleum and mineral oils [PCD 18: Natural and Synthetic Fragrance Materials]

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IS : 326 (Part 15) - 1984

Indian Standard (Reaffirmed 2000)

METHODS OF SAMPLING AND TEST FOR NATURAL AND SYNTHETIC PERFUMERY MATERIALS

PART 15 DETECTION OF PETROLEUM AND MINERAL OILS

(Second Revision)

Natural and Synthetic Perfumery Materials Sectional Committee, PCDC 18; Methods of Test Subcommittee, PCDC 18 : 1 [Ref : Doc : PCDC 18 (191) F]

1. Scope — Prescribes the method for detection of petroleum and mineral oils in natural and synthetic perfumery materials.

2. Detection of Petroleum and Mineral Oils

2.1 *General* — The saturated paraffinic hydrocarbons, found in the petroleum oils, are chemically very inert; they are not destroyed by fuming sulphuric acid. Other compounds are attacked, giving rise to reaction products which are soluble in sulphuric acid. The material has to be added to the fuming sulphuric acid very cautiously since too rapid an addition of perfumery materials is apt to cause the liberated sulphur dioxide to carry part of the acid and material out of the flask. Since petroleum fractions often contain aromatic and unsaturated compounds as well as paraffins, the separation of the paraffinic portion described above does not usually represent the total amount of added petroleum. In general the portion thus separated is usually a small percentage of the adulterant. The test may be rendered more sensitive by preliminary fractionation of the material.

The addition of petroleum fractions to a material causes a lowering of the specific gravity, refractive index and optical rotation. The solubility of the material usually is also affected.

2.2 *Apparatus*

2.2.1 *Cassia Flask*

2.3 *Reagents*

2.3.1 *Fuming Sulphuric Acid* — 15 percent oleum [*see* IS : 1089-1973 'Specification for oleum, technical (*first revision*)'].

2.3.2 *Concentrated Sulphuric Acid* — [*see* IS : 266-1977 'Specification for sulphuric acid (*second revision*)'].

2.4 *Procedure* — Place 20 ml of fuming sulphuric acid (15 percent oleum in a dry Cassia flask of approximately 150 ml capacity, and cool thoroughly in an ice-salt mixture. Cautiously add 5 ml of the material from a small burette, drop by drop, with frequent shaking and cooling in the ice-salt mixture. After the material has been added, again shake the flask and allow it to stand at room temperature for 10 minutes. Warm on a steam-bath for 5 minutes with frequent agitation, cool to room temperature, then fill to the mark with concentrated sulphuric acid and allow the flask to stand overnight, when the mineral oil will rise into the neck and separate as a colourless or straw-coloured liquid. To confirm the presence of mineral oil, remove a small amount of the separated mineral oil from the Cassia flask, using a glass tube drawn out to a small tip, and determine its refractive index. For petroleum products and mineral oils, the refractive index is expected to be less than 1·440 0.

EXPLANATORY NOTE

This standard was first published in 1952 entitled as 'Methods of test for essential oils' and subsequently revised in 1968 as 'Methods of sampling and test for natural and synthetic perfumery materials'. Taking cognizance of the need of to incorporate instrumental methods of analysis now in vogue and to facilitate inclusion of additional test methods or changes in the existing test methods and also to align these test methods with the corresponding ISO standards, the concerned Committee decided to revise and split the standard and publish individual test methods as separate parts of the original standard.

In order to facilitate proper reference to method of test given in this revised part of IS : 326, Amendment No. 3 is being issued separately to the relevant clause of IS : 326-1968 'Methods of sampling and test for natural and synthetic perfumery materials (*first revision*)' which would no longer be in force with the publication of this revision. The test method as given in this revised part will supersede the test method as given under **16.2** of IS : 326-1968.

Petroleum and mineral oils are often present as impurities and/or sepecially adulterated in the perfumery materials. Apart from detecting these through the changes in the physical properties of the material, the Committee considered it necessary to have a separate test method for the detection of petroleum and mineral oils.

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